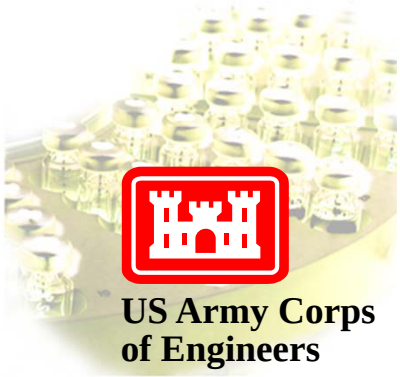
How Laboratory Sampling Techniques and Extraction Methods Affect Reproducibility of PAH Results



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USACE SI Project

- Metals and PAHs
- Significant Skeet Fragments
- ISM in the field
- Assess extraction efficiency for PAHs
- Determine Reproducibility
  - Subsampling prior to grinding (UG)
  - Subsampling after grinding (G)
  - Methods 3540C and 3550C

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Significant Skeet Fragments

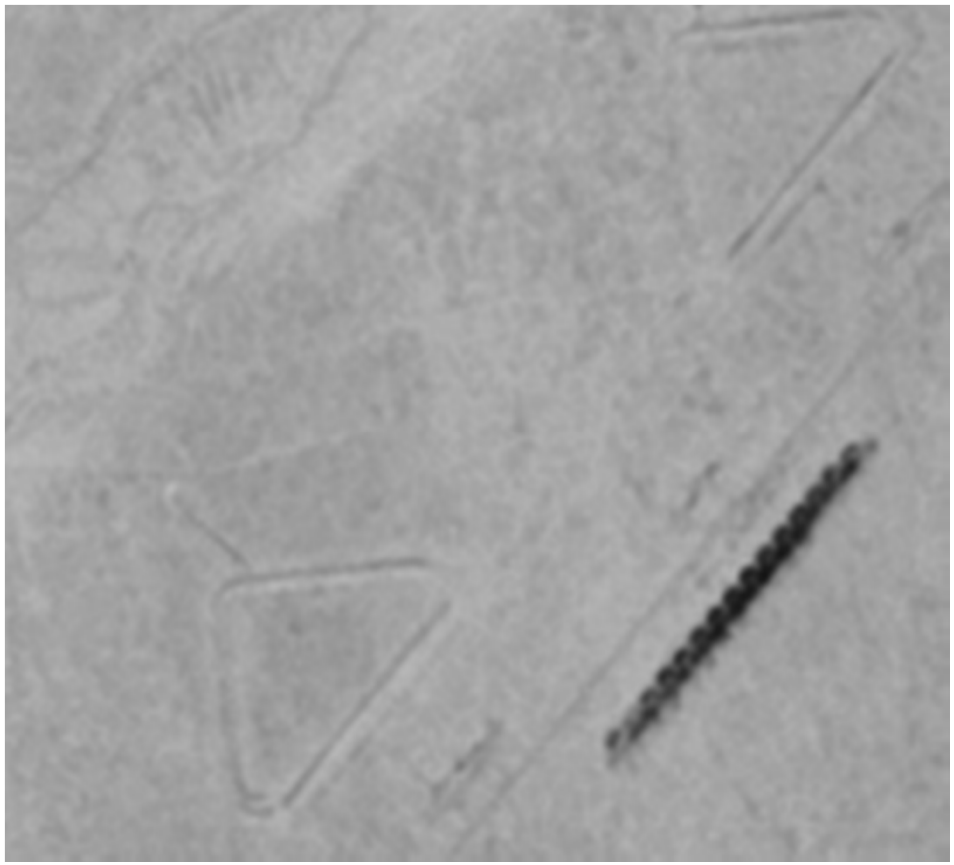

Photo: 1954, 7 years after last training

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Extraction Efficiency

- High concentration of skeet in the sample
- Dried and sieved the sample
  - 24 hour drying time
  - Mortar and Pestle (breakup large fraction)
  - Skeet crushes to a fine powder
  - # 10 Sieve
- Soxhlet Serially extracted 5 times
- Sonication Serially extracted 5 times

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Sieved Skeet (#10)

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Non Passing Portion (rocks and sticks)

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Original Extraction Soxhlet

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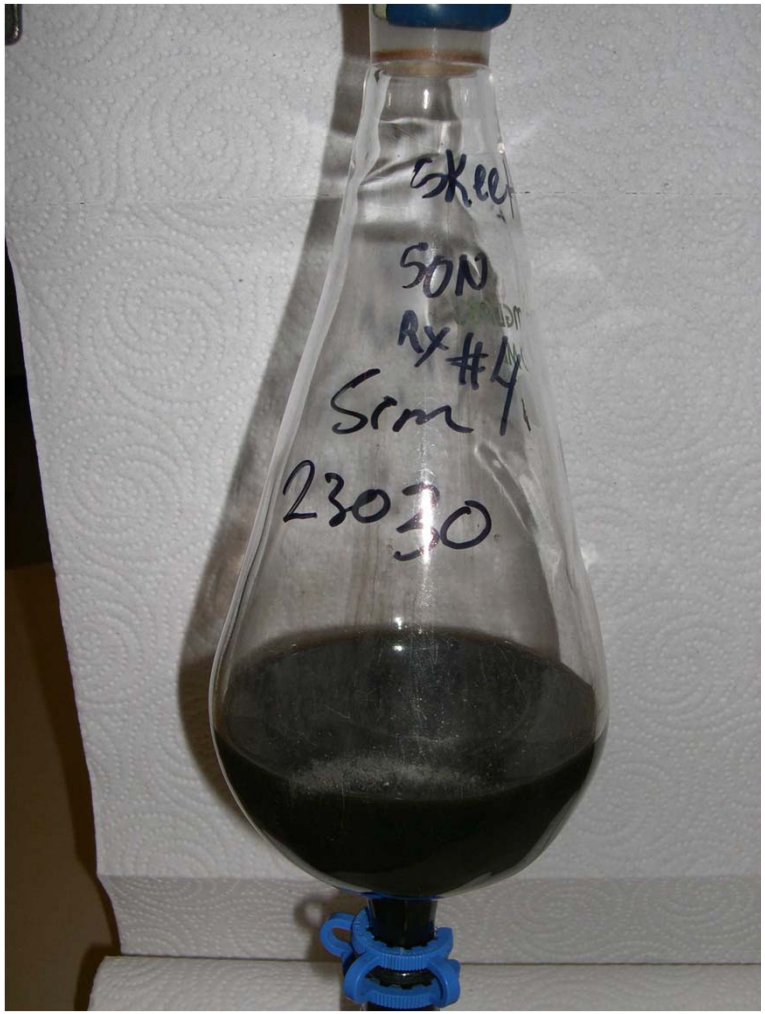
4th Serial Extraction by Sonication



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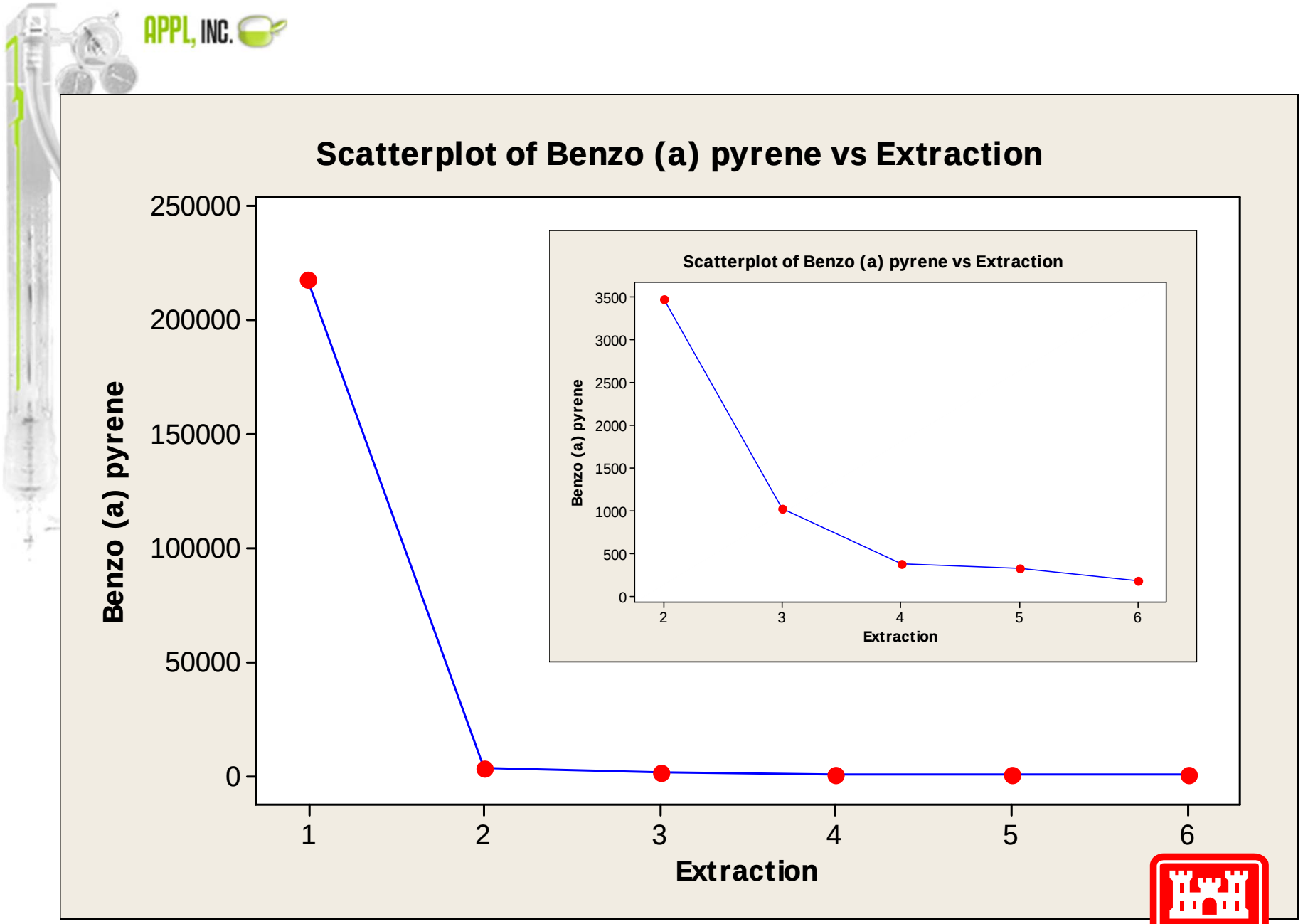

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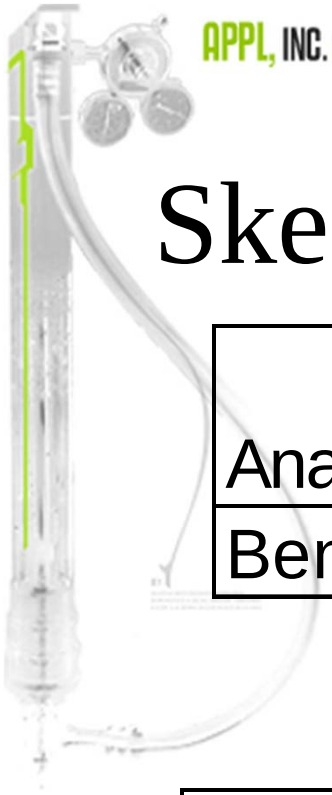

Skeet Serial Extraction in mg/Kg

Analyte	Original Extraction	RX 1
Benzo (a) pyrene	2300	10.0

Analyte	RX 2	RX 3	RX 4	RX 5
Benzo (a) pyrene	1.28	0.39	0.24	0.17

Soil Screening Level for Benzo (a) pyrene: 50 ug/Kg

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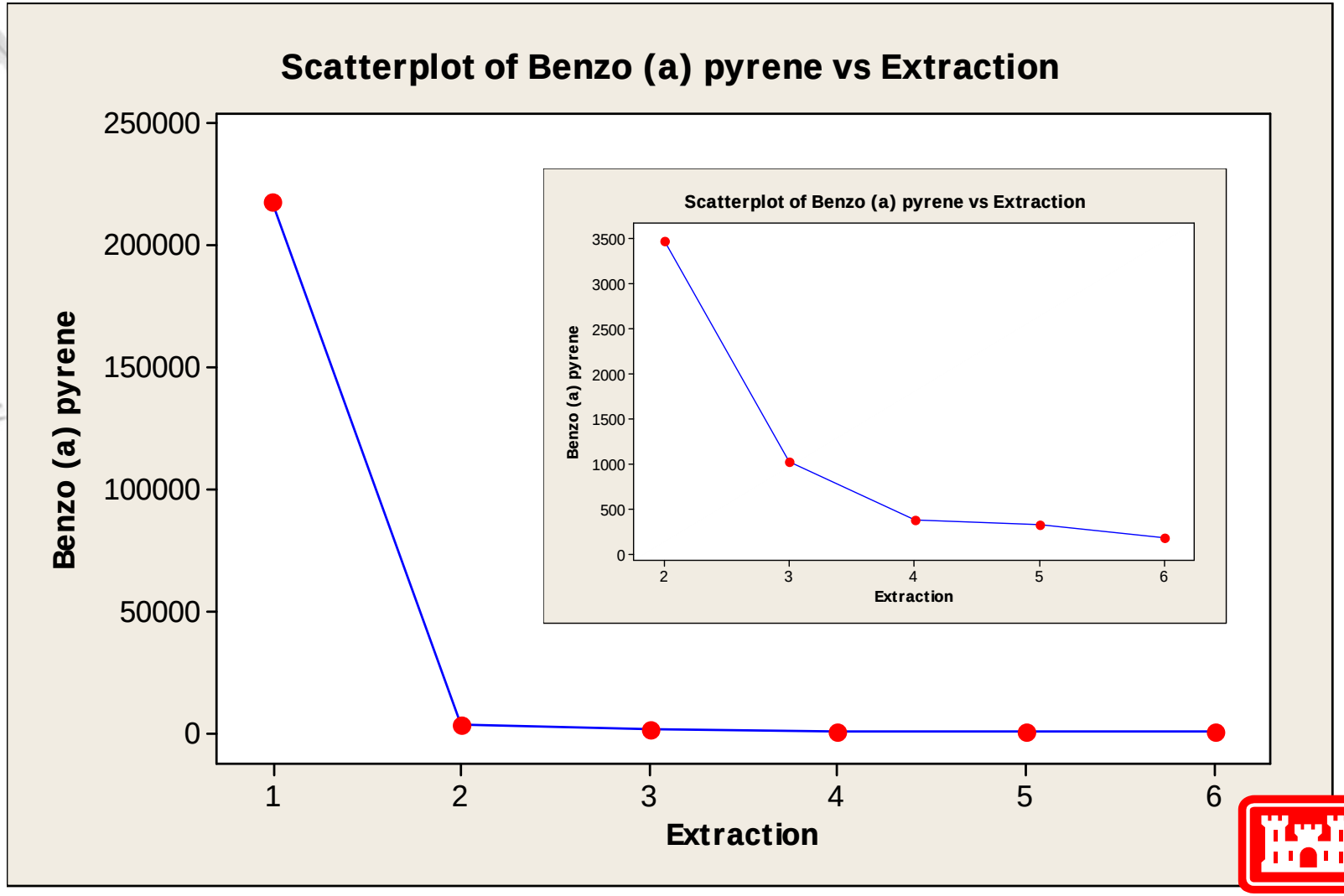
Compound	Original to RX 1	RX 1 to RX 2	RX 2 to RX 3	RX 3 to RX 4	RX 4 to RX 5
Benzo (a) pyrene	0.44%	12.8%	30.2%	63.5%	70.5%



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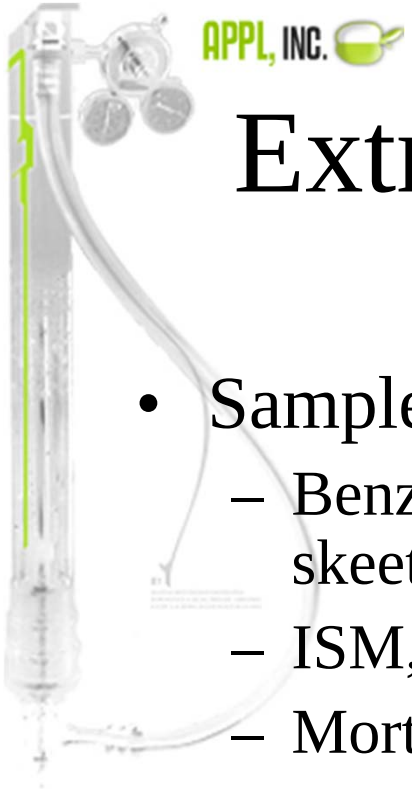

Extraction Efficiency in Field Sample

- Sample A
  - Benzo (a) pyrene concentrations 10 times less than the skeet fragment sample concentration
  - ISM, 30 meters x 30 meters,100 increments
  - Mortar and Pestle manual grinding to pass #10 sieve
- Sample B
  - Benzo (a) pyrene 100 times less than the skeet fragment sample concentration
  - ISM, 30 meters x 30 meters,100 increments
  - Mortar and Pestle manual grinding to pass #10 sieve

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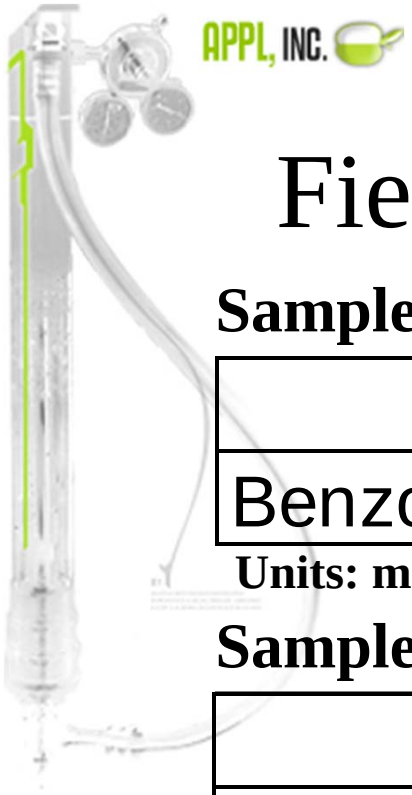

Field Sample Serial Extraction

Sample A

Analyte	Original	RX 1
Benzo (a) pyrene	116	0.136

Units: mg/Kg

Sample B

Analyte	Original	RX 1
Benzo (a) pyrene	22.0	0.007

Units: mg/Kg

Extraction Efficiency

Analyte	Sample A	Sample B
Benzo (a) pyrene	0.12%	0.03%

Extraction Method: Soxhlet 3540C

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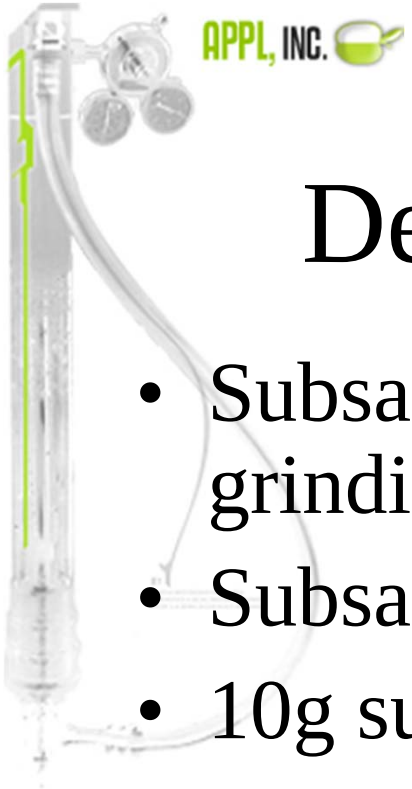

Determine Reproducibility

- Subsampling (8330B App. A) prior to grinding
- Subsampling (8330B App. A) after grinding
- 10g subsample aliquots (high level)
- Methods Soxhlet 3540C and Sonication 3550C
- Custom PAH PE Samples (ERA),
- Field Samples (~ 1 ppm Benzo (a) Pyrene)

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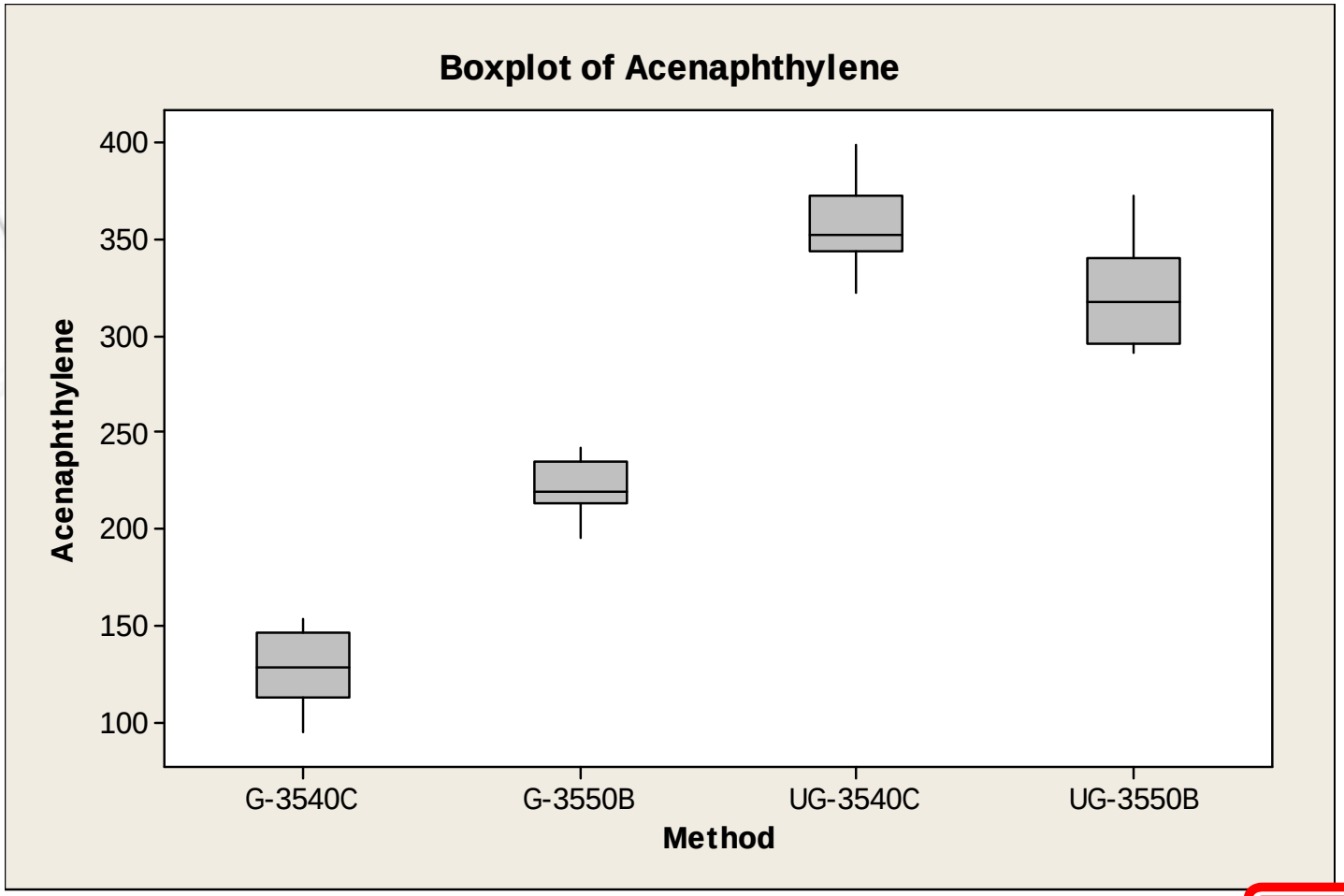
PE Sample Reproducibility



Laboratory Replicates = 10 (n = 10)

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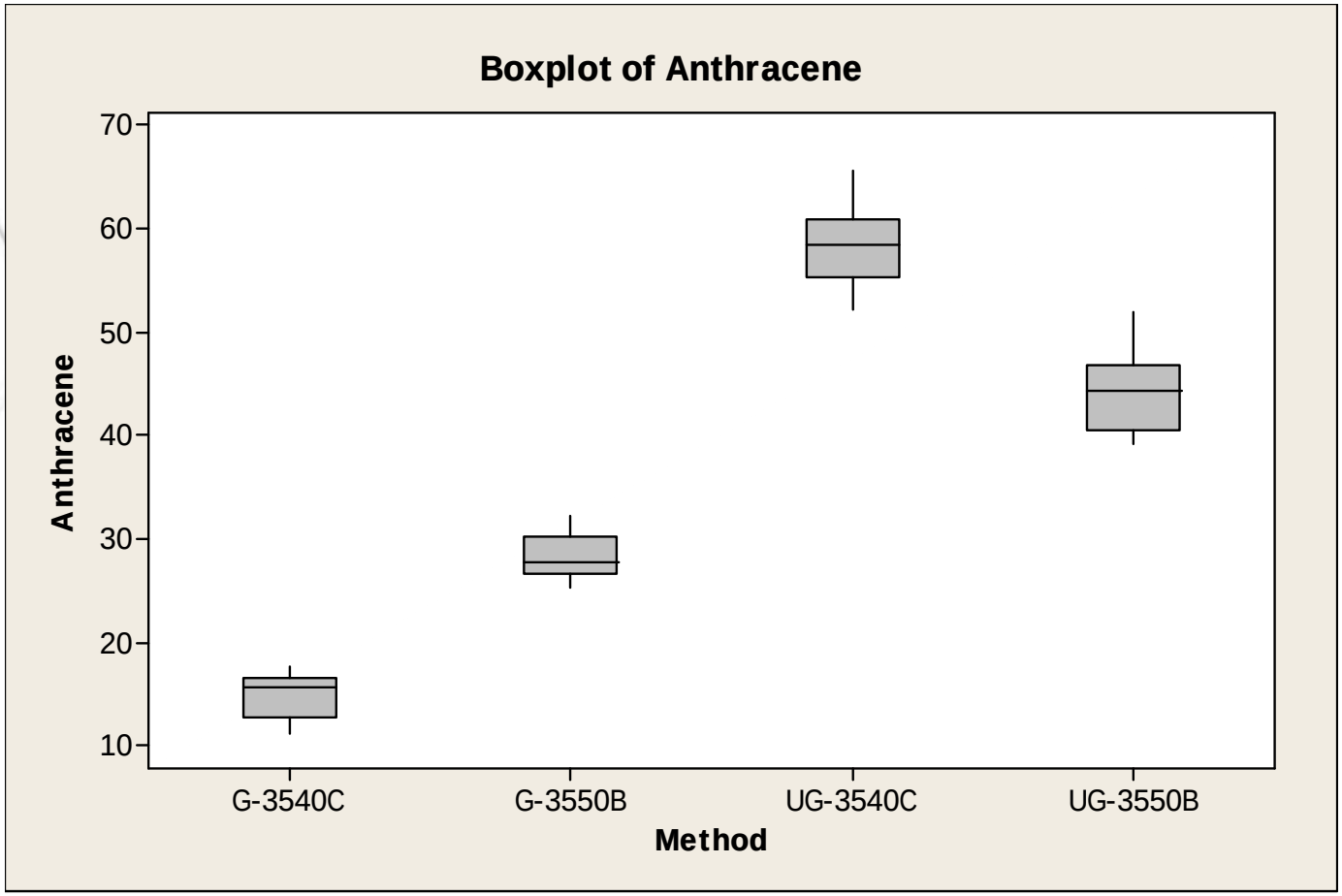
PE Sample Reproducibility



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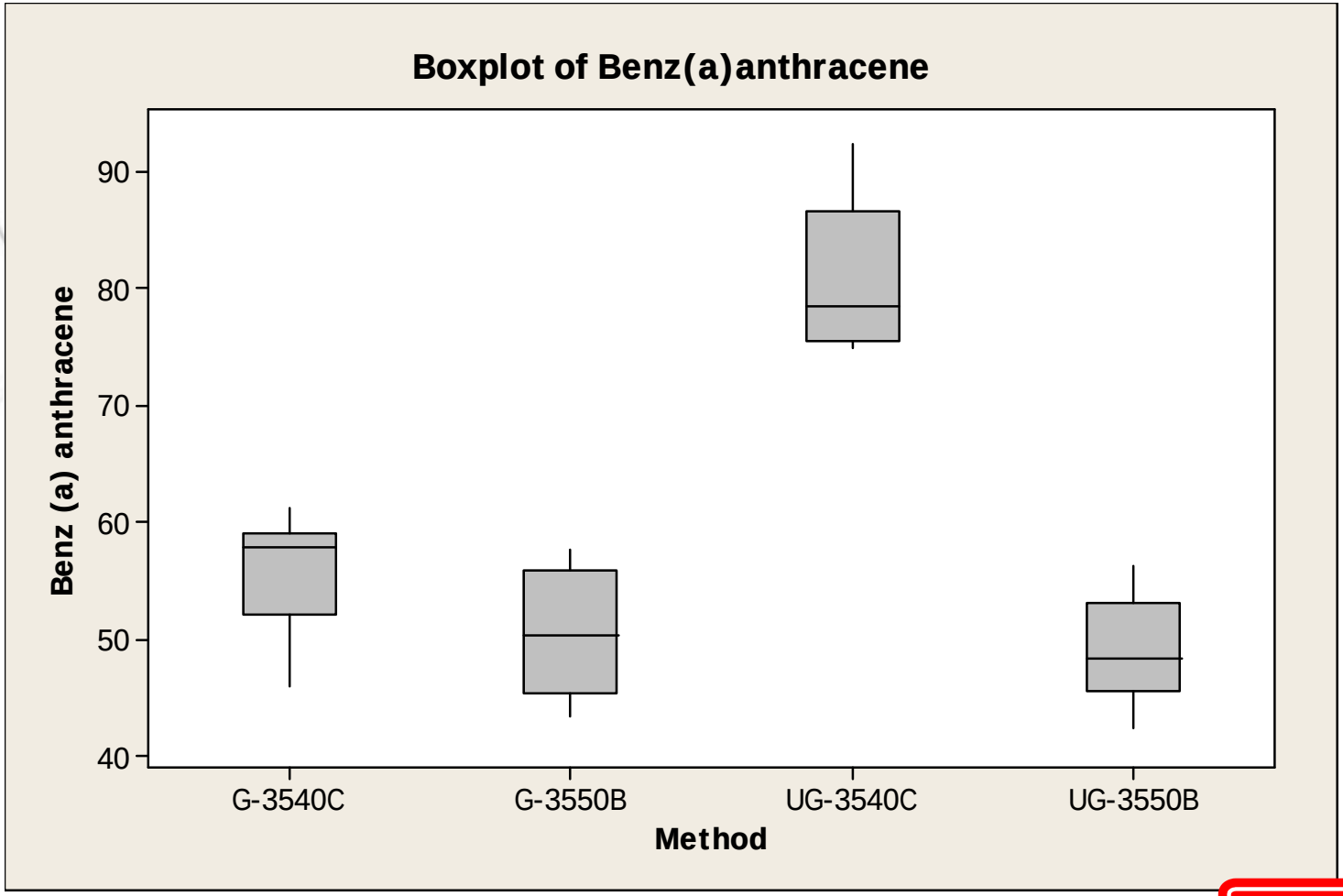
PE Sample Reproducibility



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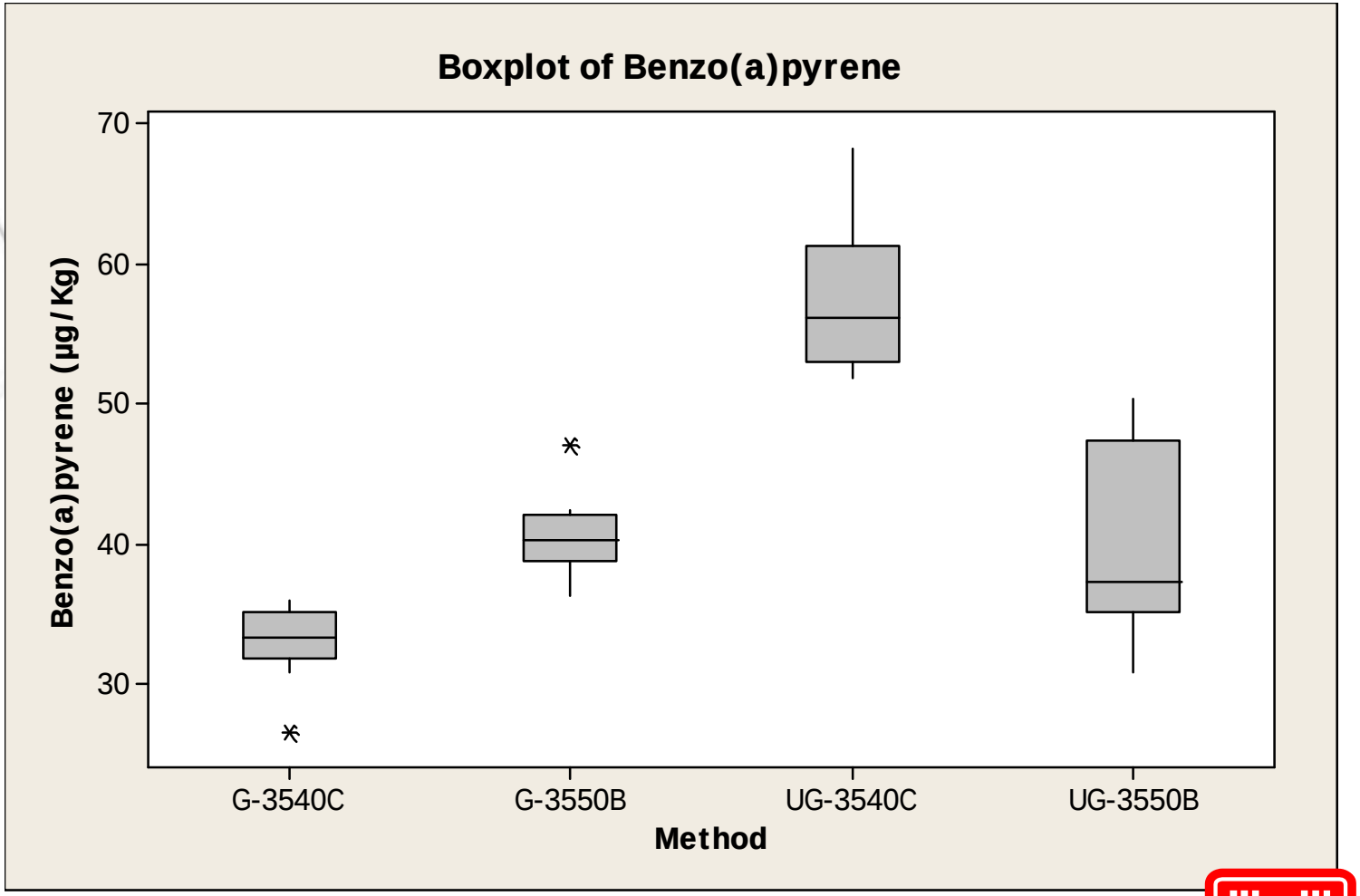
PE Sample Reproducibility



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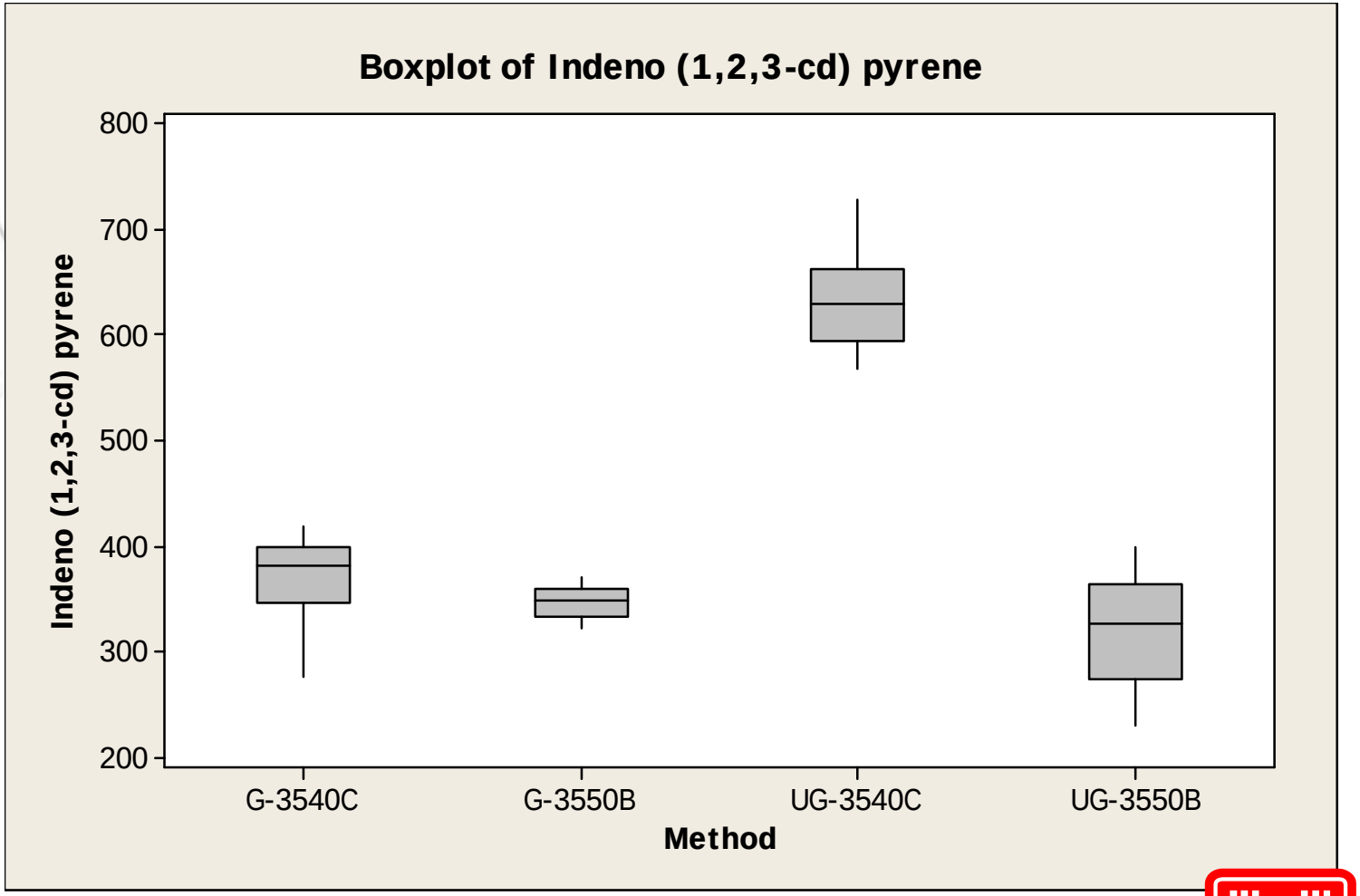
PE Sample Reproducibility



Laboratory Replicates = 10 (n = 10)

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Sonication Extraction PE Results

Analyte	% R Unground	% R Ground	% Lower control limit	% Upper control limit
2-Methylnaphthalene	36.2	36.1	33.5	110
Acenaphthylene	42.3	29.4	15.5	80.3
Acenaphthene	44.6	40.7	25.3	103
Fluorene	47.3	47.0	31.5	107
Phenanthrene	49.7	52.0	42.1	116
Anthracene	43.8	27.9	21.5	80.9
Fluoranthene	49.7	53.6	45.1	119
Pyrene	50.4	53.2	37.3	125
Benz (a) anthracene	48.3	50.2	36.6	109
Chrysene	47.3	51.1	44.9	122
Indeno (1,2,3-cd) pyrene	37.5	40.8	37.7	115
Benzo (b) fluoranthene	118	116	41.3	124
Benzo (k) fluoranthene	85.8	84.6	41	112
Benzo (a) pyrene	39.9	40.5	24.5	102
Dibenz (a,h) anthracene	49.3	52.8	39.1	115
Benzo (g,h,i) perylene	42.9	45.8	33.7	123

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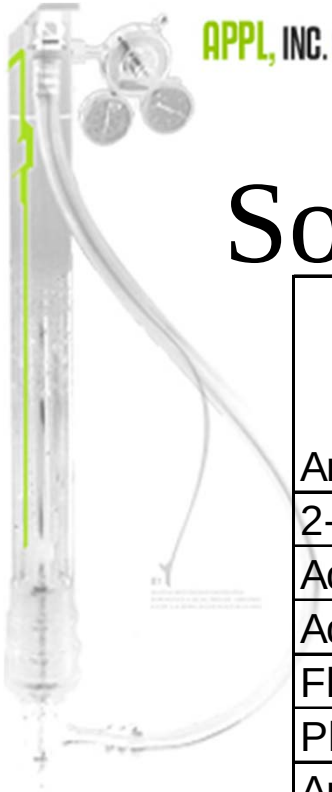

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Soxhlet Extraction PE Results

Analyte	%R Unground	%R Ground	% Lower Control Limit	% Upper Control Limit
2-Methylnaphthalene	41.3	28.3	33.5	110
Acenaphthylene	47.1	16.9	15.5	80.3
Acenaphthene	50.8	33.2	25.3	103
Fluorene	64.0	45.4	31.5	107
Phenanthrene	72.2	58.2	42.1	116
Anthracene	57.8	14.8	21.5	80.9
Fluoranthene	77.0	57.3	45.1	119
Pyrene	69.0	53.7	37.3	125
Benz (a) anthracene	80.6	55.1	36.6	109
Chrysene	62.8	52.2	44.9	122
Indeno (1,2,3-cd) pyrene	74.1	43.3	37.7	115
Benzo (b) fluoranthene	73.7	132	41.3	124
Benzo (k) fluoranthene	73.3	96.2	41.0	112
Benzo (a) pyrene	91.9	32.9	24.5	102
Dibenz (a,h) anthracene	104	57.0	39.1	115
Benzo (g,h,i) perylene	70.4	47.8	33.7	123

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Field Sample UG vs G

- Dried to constant weight (24 hours)
- Mortar and Pestle Manual Grinding
- Sieved using #10
- Subsampled (8330b App. A) prior to Puck Mill grinding (UG)
- Ground using Puck Mill (5 x 60s)
- Subsampled after grinding (G)

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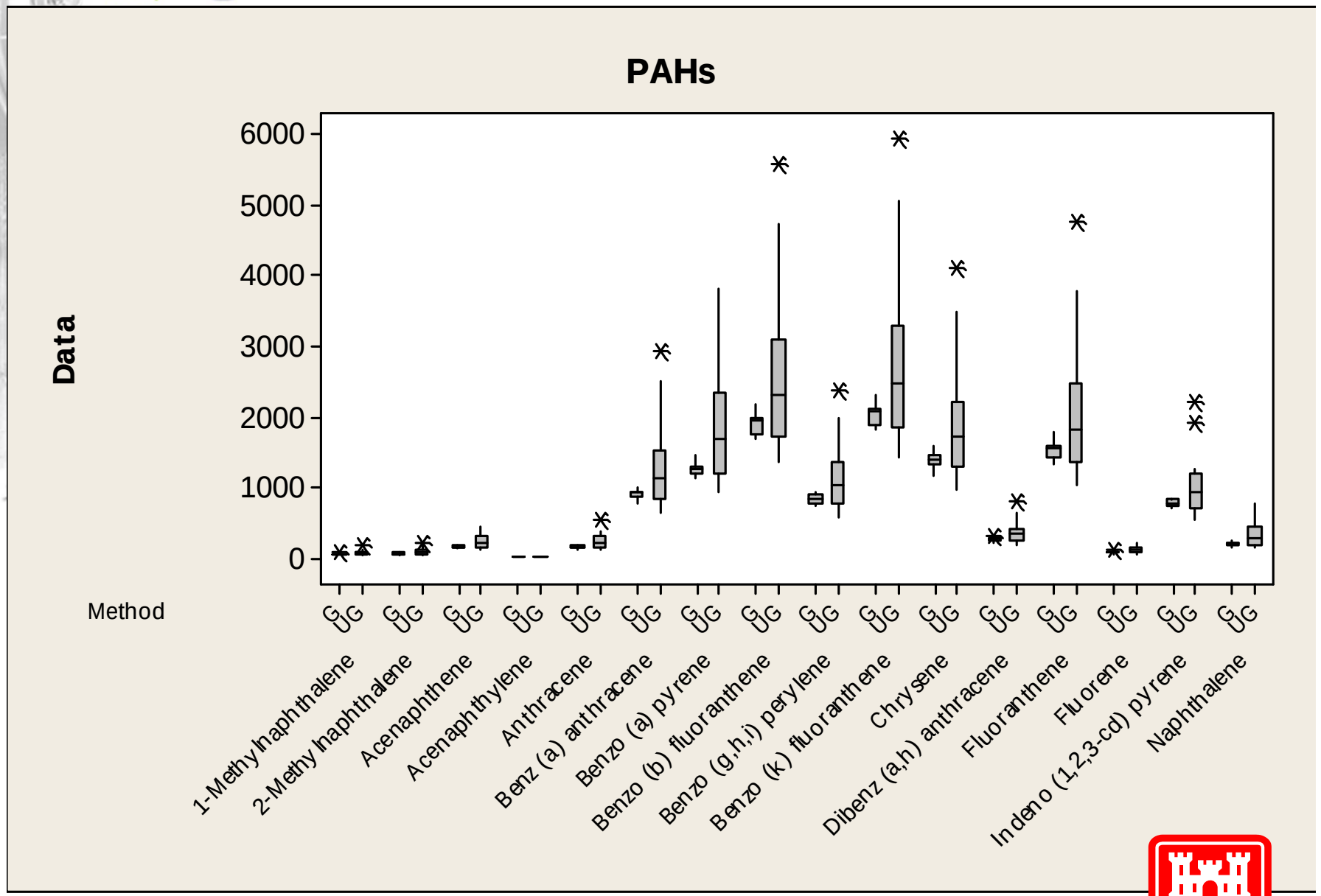

Laboratory Replicates = 16 (n=16)

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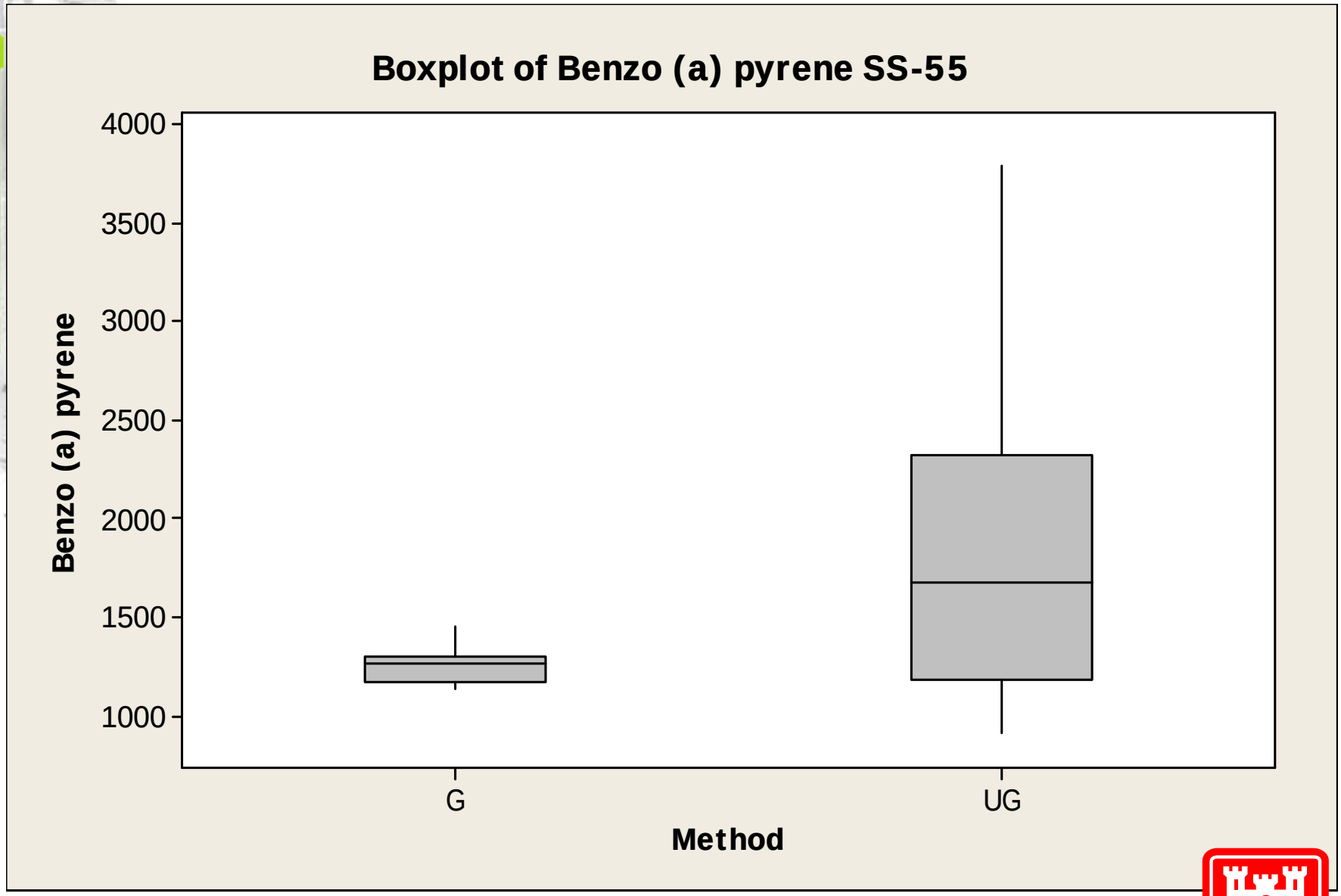

Laboratory Replicates = 15 (n=15)

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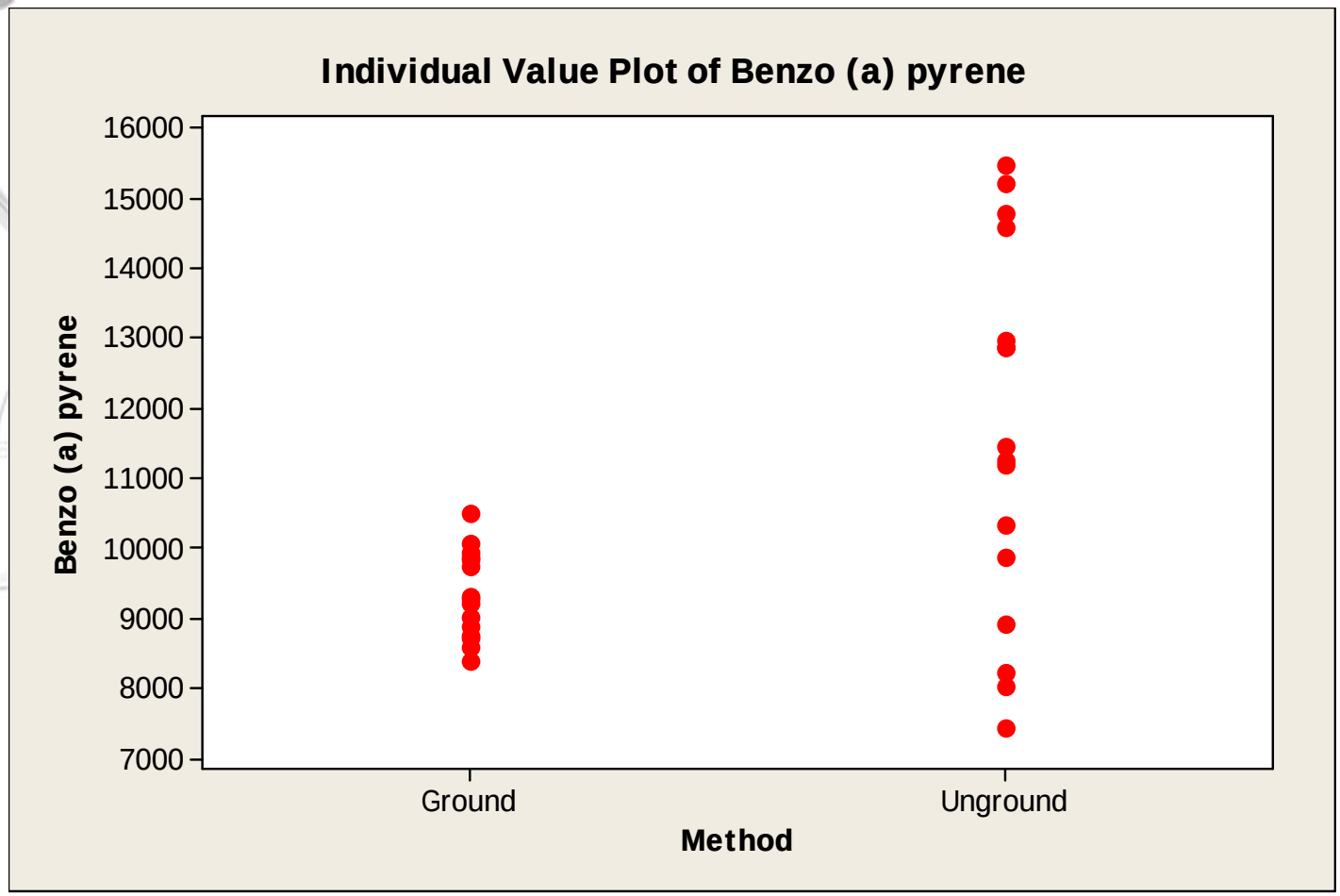

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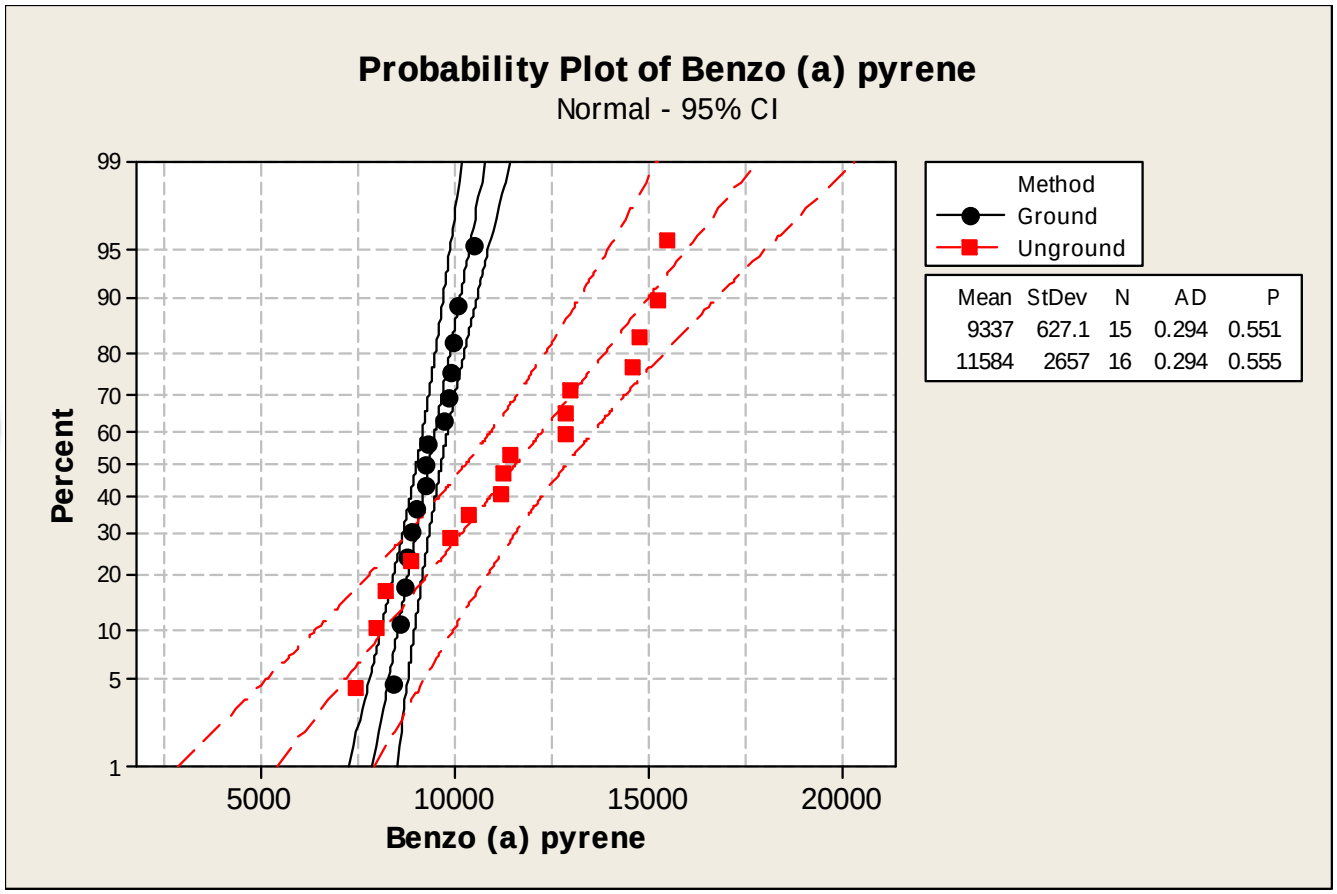

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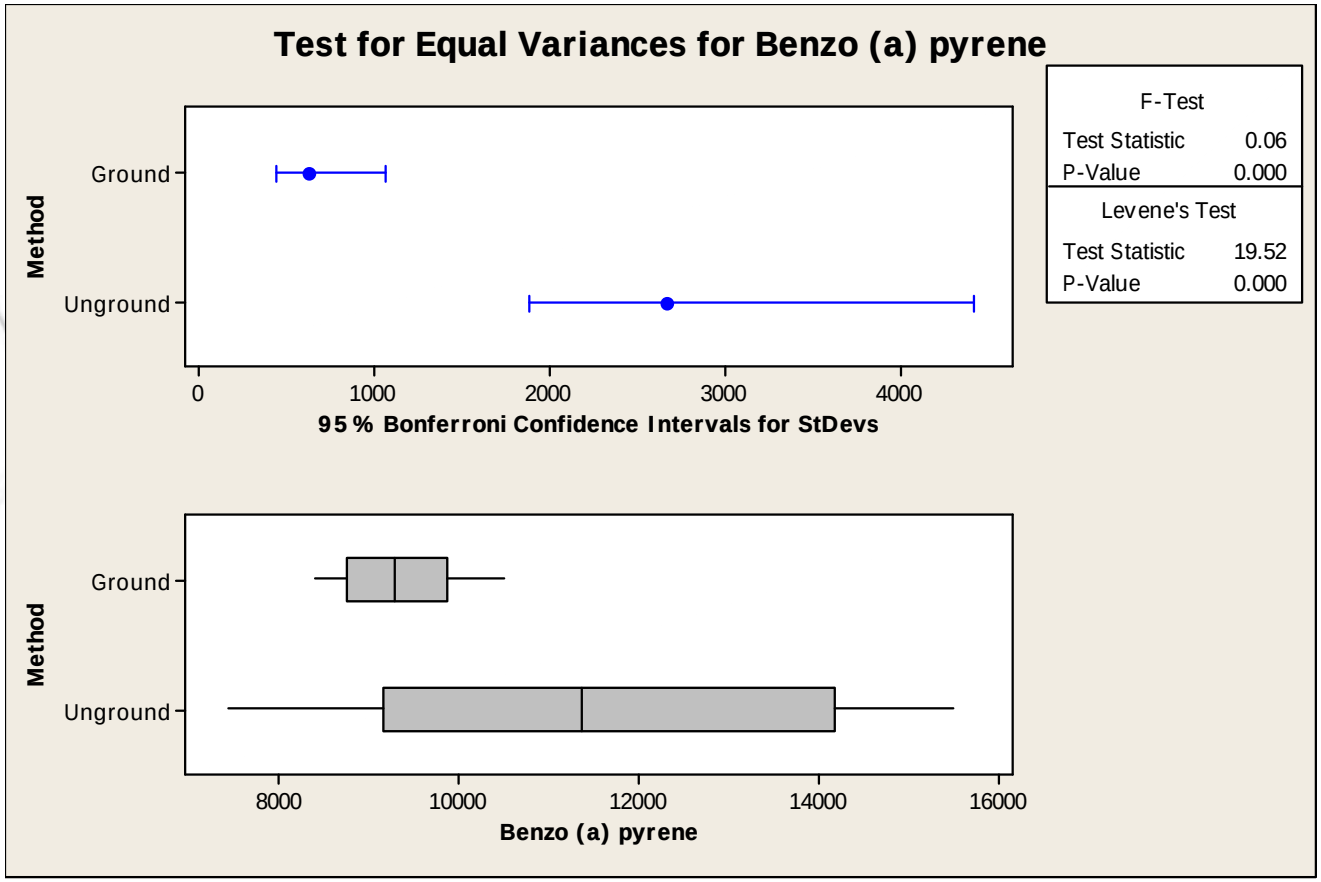

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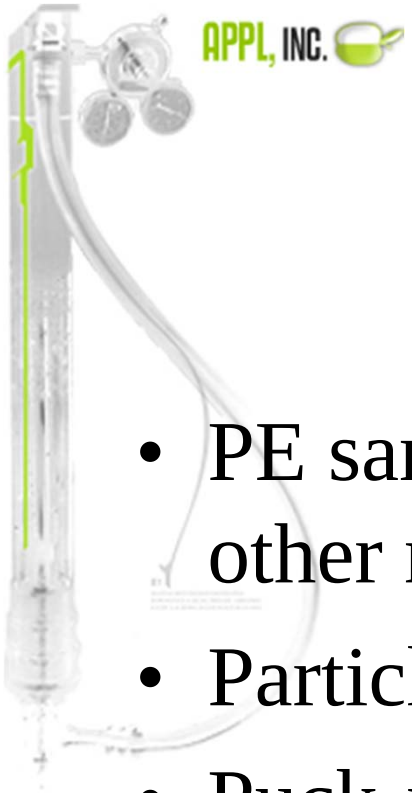

Conclusions

- PE sample needs to be researched using other mechanical grinding techniques
- Particle size impacts to Soxhlet extraction?
- Puck-mill grinding produces a low bias for PAH analyses
- Need to look at alternative grinding techniques

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Full Disclosure

- All statistical representations of this data were prepared by:
  - Dr. Thomas Georgian USACE, EM-CX
- If you are in disagreement as to their relevance, statistical significance or just don't like the background color please take it up with TOM!
- All study design failings are the fault of Brian Jordan and his indecipherable instructions

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Questions???

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